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ULTRASONIC DETECTION OF OXYGEN IN TITANIUM  
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# ULTRASONIC DETECTION OF OXYGEN IN TITANIUM ALLOY PLATES AND WELDMENTS

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## ABSTRACT

Dissolved oxygen contamination during GTA and GMA welding of titanium alloys can result in severe embrittlement of the weld region. In order to evaluate the feasibility of ultrasonic testing for quantitatively detecting the presence of interstitial gas contamination in weldments of Ti-6211, ultrasonic wave velocity and ultrasonic attenuation measurements were performed on a series of five specimens with nominal oxygen levels of 0.07, 0.14, 0.20, 0.24 and 0.29 percent by weight. Density measurements, in addition to the ultrasonic wave velocity data, enabled relative determination of elastic moduli. Variations in the ultrasonic data were correlated with results from scanning electron microscopy and hardness testing.

## INTRODUCTION

Titanium alloys exhibit high strength-to-weight ratios and good corrosion resistance and are, therefore, desirable for use in structural applications. The safe, in-service use of titanium alloy weldments in critical, load-bearing parts requires the development of reliable nondestructive methods for inspecting the mechanical integrity of the weld region.

Unfortunately, current nondestructive evaluation technology has proven inadequate for such alloys and applications. Since one of the primary nondestructive inspection techniques is ultrasonics, it is expedient to determine the usefulness and limitations of ultrasonic inspection for the materials in question. This requires careful acoustical characterization of these materials in order that appropriate accept/reject criteria be established.

The mechanical properties of titanium alloys are known to be sensitive to oxygen content (1-26). Above a certain threshold concentration oxygen appears to have a severe embrittling effect on titanium alloys (6-8, 17, 20-26). Since titanium alloys subjected to gas tungsten-arc (GTA) and gas metal-arc (GMA) welding techniques are susceptible to dissolved gas contamination and the resulting embrittlement, it is of interest to investigate methods for nondestructively determining the oxygen content of a titanium alloy welded joint. Pure titanium has a hexagonal close-packed structure (alpha phase) at room temperature, transforms to a body-centered cubic structure (beta phase) at 883°C, and melts at 1668°C. In this investigation the alloy used was titanium 6211, which has a nominal composition of 6 weight percent aluminum, 2 weight percent columbium, 1 weight percent molybdenum, and 1 weight percent tantalum with the balance ideally being pure titanium. This alloy of structural importance is classed as a near alpha alloy because it contains primarily the alpha phase with small amounts of beta phase material interspersed.

The objective of the present research was to evaluate the feasibility of using ultrasonics for quantitatively assessing the presence of oxygen gas contamination in weldments of titanium 6211. To achieve this goal, ultrasonic wave velocity and attenuation measurements were made on a series of titanium 6211 specimens possessing varying oxygen contents. The measured variations in ultrasonic data were compared with the results of scanning electron microscopy and hardness tests.

### BACKGROUND

Ultrasonic wave propagation analysis of a material is a nondestructive evaluation technique that provides information about the elastic properties and absorption characteristics of the material in which the wave propagates. Absolute measurements of shear and longitudinal wave speed can be used to calculate useful material parameters, such as the effective Young's modulus and the effective shear modulus. The term "effective modulus" refers to the fact that the modulus is calculated for a material that is assumed to be linear elastic, homogeneous, and isotropic, which is a fair approximation for some fine-grained polycrystalline materials. The energy loss, or ultrasonic attenuation of elastic waves propagating in a solid, may be divided into contributions from geometrical and intrinsic effects. Of interest here are the intrinsic effects which include scattering of the ultrasonic wave by inhomogeneities, conversion of sound energy to heat as a result of elastic deformation, interaction with thermal phonons, and dislocation

damping (27).

Since the concentration of an alloying element, oxygen in this case, affects the same set of variables that determine ultrasonic wave propagation characteristics, it is expected that ultrasonic techniques will be suitable for determining oxygen concentration in contaminated Ti-6211 specimens. It has been shown that foreign solute atoms invariably change elastic moduli (5, 10, 11, 18). Additionally, solute atoms can act as pinning points for dislocations and would, therefore, be expected to influence attenuation measurements. Some previous studies of the effects of interstitial gases on elastic wave propagation also indicate the feasibility of this method for determination of dissolved gas concentration. Hsu and Conrad (18) looked at the effect of oxygen on the elastic properties of titanium-oxygen alloys through ultrasonic wave velocity measurements performed on specimens with oxygen content in the range of 0.04 weight percent to 2 weight percent and controlled impurity content. It was found that both density and longitudinal wave velocity increased with increasing oxygen content. Since the relative change in the wave velocity was greater than that in the density, the dynamic elastic modulus was increased by the oxygen in solid solution. Ultrasonic attenuation was also observed to increase with increasing oxygen content. The authors concluded that it was likely that the interstitial oxygen atoms in the titanium lattice actually increased the binding forces between atoms.

The influence of oxygen content on the internal friction

characteristics of titanium, particularly the modification of grain boundary relaxation phenomena was investigated by Pratt, Bratina, and Chalmers (10) with the objective of obtaining further information concerning the exact role of the interstitial solutes in alpha titanium. In the oxygen-containing alloys, the internal friction peak characteristic of grain boundary slip occurred at higher temperatures than in pure titanium and the heat of activation increased. The presence of oxygen resulted in an additional small peak not observed in the relaxation spectra of pure titanium. They also found that the addition of 4.5 atomic percent oxygen raised the rigidity modulus, which is consistent with the results of Hsu and Conrad (18).

## EXPERIMENTAL PROCEDURES

### Metallography

Metallographic specimens were cut from the oxygen contaminated Ti-6211 samples. The specimens were ground on successively finer silicon carbide papers down to 600 grit and then polished on lapidary wheels using 15 micron and 0.05 micron compounds. A two step etching technique was used to reveal microscopic detail. In the first step, the polished specimen was briefly swabbed with a 2 ml HF, 98 ml water solution. This etched the alloy and stained the alpha phase. The second step, swabbing with a 1 ml HF, 2 ml HNO<sub>3</sub>, 97 ml water solution, removed the stain, leaving a light field of alpha phase material in which the beta phase appeared as finely dispersed dark lines when viewed through an optical



microscope. When viewed through a scanning electron microscope (SEM), the beta phase appeared as finely dispersed light lines in a dark field of alpha phase material.

### Ultrasonic Measurements

Flat parallel faces were machined on each of the oxygen contaminated specimens prior to performing both ultrasonic measurements and hardness measurements. A high degree of parallelism of specimen faces was required in order to minimize errors in the measured attenuation values caused by specimen wedging effects. The ultrasonic wave velocity and attenuation measurement system used in the present research is shown schematically in Fig. 1. Conventional pulse-echo overlap techniques as described by Chung, Silversmith, and Chick (28) were used to measure the longitudinal wave velocities. Attenuation measurements were made with an automatic attenuation recorder, which includes a time gate permitting selection of any two echoes from the received wave train, an automatic gain control to stabilize the amplitude of the first echo, and circuitry to obtain the logarithm of the ratio of two selected echo amplitudes and display the result in decibels.

Commercial, longitudinal wave, ceramic transducers along with appropriate couplants were used in this research. An Aerotech couplant (a light oil) was used for the wave velocity measurements. A 2 mm thick elastomer coating (developed by Martin Marietta Laboratories in Catonsville, MD) was applied to a 2.25 MHz, longitudinal wave, commercial transducer and was used to acoustically couple the transducer to the oxygen

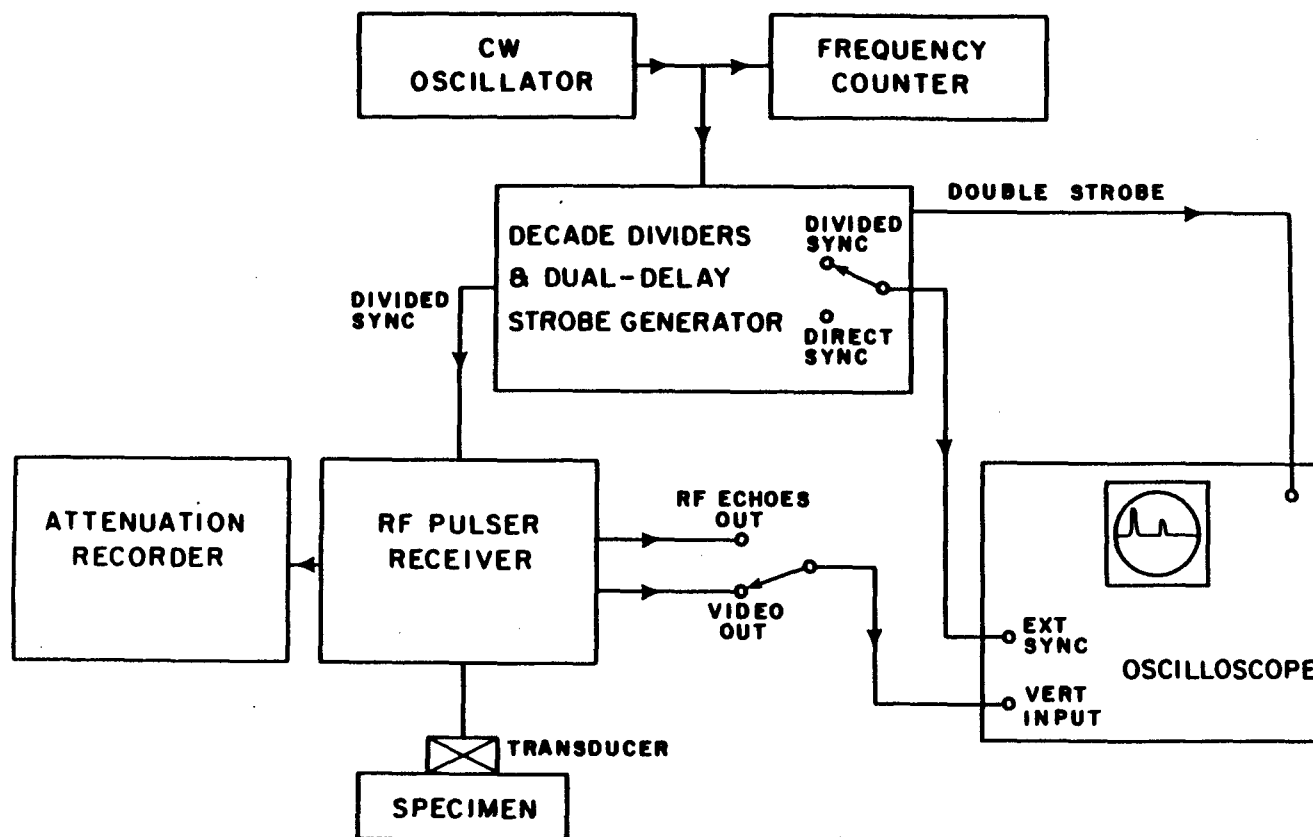


Fig. 1. Ultrasonic Wave Velocity and Attenuation Measuring System

contaminated specimens for the attenuation measurements. The viscoelastic properties of this coating make it an ideal couplant for attenuation measurements. When subjected to low frequency stress cycles (low rates of deformation), the elastomer is flexible and can be forced down on a sample for a reproducible acoustical couple. When subjected to high frequency stress cycles (high rates of deformation) the coating behaves like a rigid body. The ultrasonic pulses effectively propagated through a rigid acoustical coupling media. This elastomer coating provided more reproducible attenuation measurements than was possible with conventional couplants. The thickness of the coating made it unsuitable for accurate velocity measurements.

#### Hardness Measurements

Rockwell "C" hardness measurements were made on the oxygen contaminated Ti-6211 specimens in accordance with ASTM Standard E18. A diamond tipped "Brale" indenter with a 150 kg load was used. Since Ti-6211 continued to exhibit plastic flow after the application of the major load, the dial indicator continued to move after the operating lever stopped. For this reason the operating lever was brought to its latched position at an elapsed time of 30 seconds between application and removal of load.

#### Density Measurements

Cubes nominally 1 inch on a side were machined from each of the oxygen contaminated Ti-6211 specimens. The volume

was measured to an accuracy of  $0.001 \text{ cm}^3$ , and the mass was measured to an accuracy of 0.001 g. Two cubes were fabricated from each of the five oxygen contaminated specimens for mass density determination.

## RESULTS AND DISCUSSION

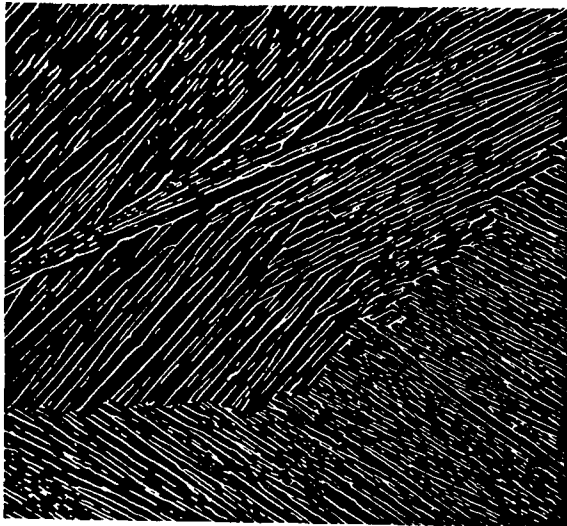
To evaluate the feasibility of ultrasonic test methods for detecting quantitatively the presence of interstitial gas contamination in weldments of Ti-6211, various measurements were performed on a series of five specimens with approximate oxygen levels of 0.07, 0.14, 0.20, 0.24, and 0.29 percent by weight. The oxygen was introduced into the molten Ti-6211 samples in the form of  $\text{TiO}_2$  powder, and the samples were subsequently beta processed (heated and rolled in the beta phase field at  $925^\circ$  in a controlled, inert atmosphere). The samples were then air-cooled. Chemical analysis of the specimens was performed by the RMI Company of Niles, Ohio, producers of the plate samples. The results of this analysis are presented in Table I.

### Metallography

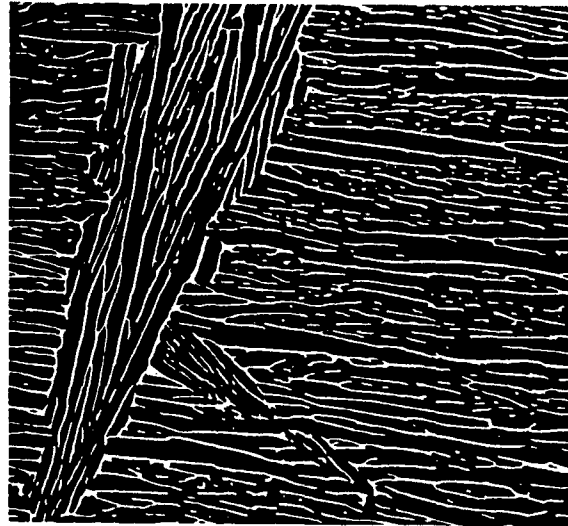
Metallographic analysis performed on the five specimens revealed significant microstructural differences. The photomicrographs (SEM) displayed in Fig. 2 show distinct changes in microstructure with increasing oxygen content. As the oxygen content increased, the alpha platelets became wider; hence, the separation of the layers of beta increased. Additionally, the beta layers thickened until they began to

Table I. Specimen Compositions

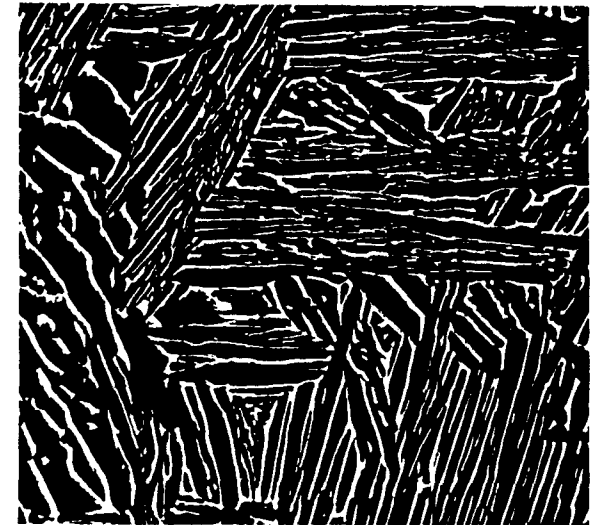
SPECIMEN	COMPOSITION (weight percent)								
	Ti	Al	Cb	Mo	Ta	C	N	Fe	O
A	90.3	6.0	1.95	0.7	0.88	0.02	0.010	0.05	0.069
B	89.9	6.0	2.09	0.9	0.97	0.02	0.006	0.03	0.136
C	90.2	5.9	1.90	0.8	0.94	0.02	0.005	0.03	0.194
D	90.1	5.8	2.05	0.8	0.99	0.02	0.006	0.03	0.238
E	89.8	5.9	2.16	0.7	1.06	0.03	0.008	0.03	0.290



SPECIMEN A  
0.069 wt% OXYGEN



SPECIMEN B  
0.136 wt% OXYGEN

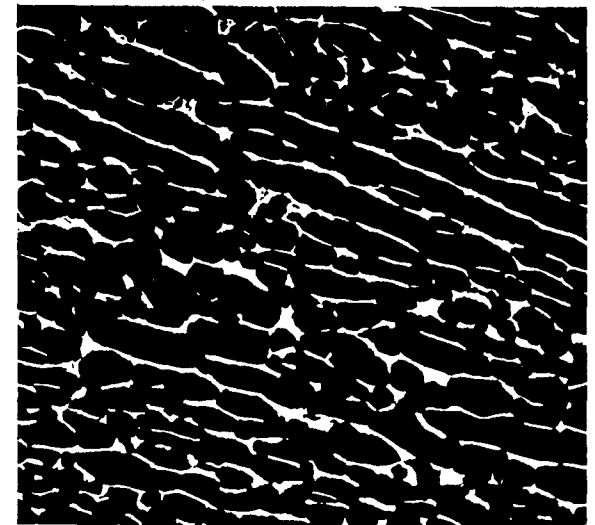


SPECIMEN C  
0.194 wt% OXYGEN



SPECIMEN D  
0.238 wt% OXYGEN

FIG. 2. SEM PHOTOMICROGRAPHS  
OF Ti-6211 PLATE WITH VARYING  
OXYGEN CONTENTS (750X)



SPECIMEN E  
0.290 wt% OXYGEN

break up and become more globular in appearance. This is especially evident in the SEM photomicrograph of specimen E, the most heavily contaminated specimen. It should be noted that the microstructures of the oxygen-contaminated plates are not representative of those observed in the weld region of welded Ti-6211 specimens.

### Density Measurements

The results of density measurements performed on this series of five oxygen contaminated specimens are presented in Fig. 3. Note that the mass density ( $\rho$ ) increases with increasing oxygen content because the oxygen enters the titanium lattice interstitially. These results are consistent with the results of Hsu and Conrad (18).

### Ultrasonic Wave Velocity Measurements

Longitudinal wave velocity ( $V_L$ ) was found to decrease with increasing oxygen content (see Fig. 4). Specimen C gave anomalous velocity results; it is suspected that this was due to improper thermomechanical preparation. Figure 5 is a plot of the average product  $\rho V_L^2$  (normalized) versus oxygen content for the five specimens. The behavior of the quantity  $\rho V_L^2$  is indicative of the behavior of the elastic moduli, so Fig. 5 shows that the elastic moduli tend to decrease with increasing oxygen content.

Discussion of the observation of a decrease in the longitudinal wave velocity requires consideration of the material properties that affect  $V_L$  in these oxygen contaminated Ti-6211

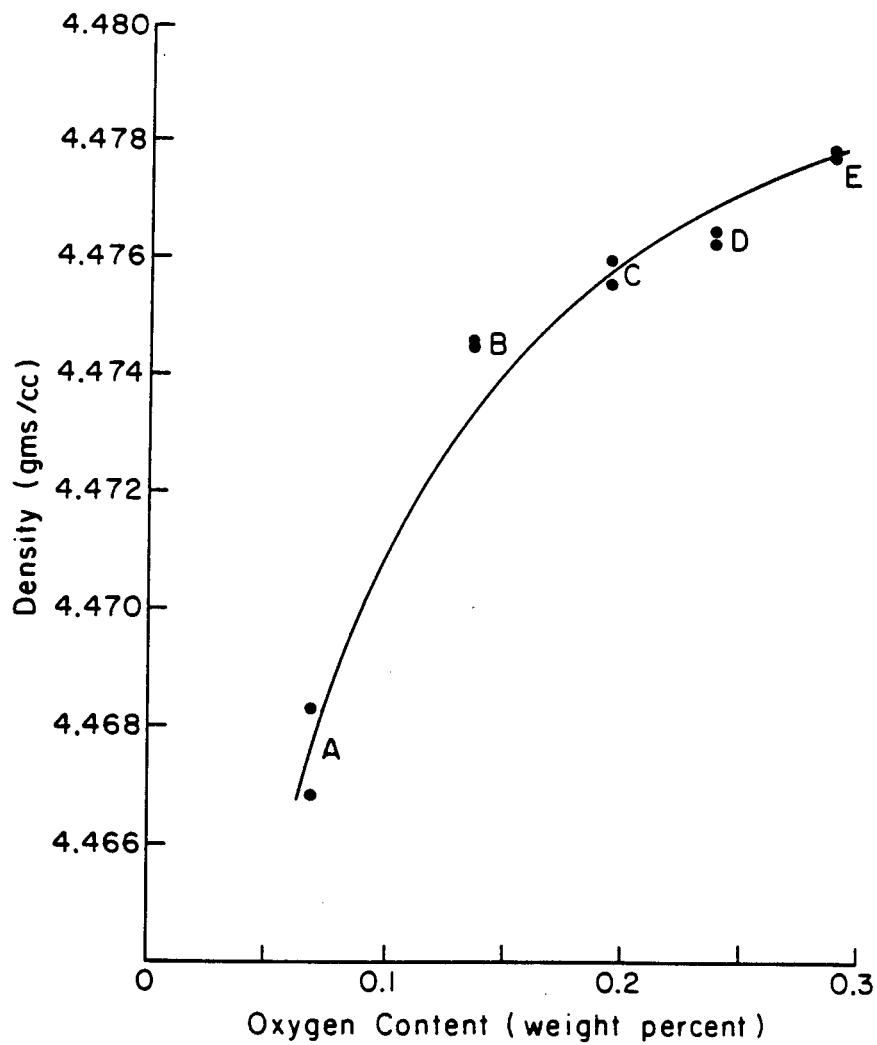


Fig. 3. Mass Density vs. Oxygen Content



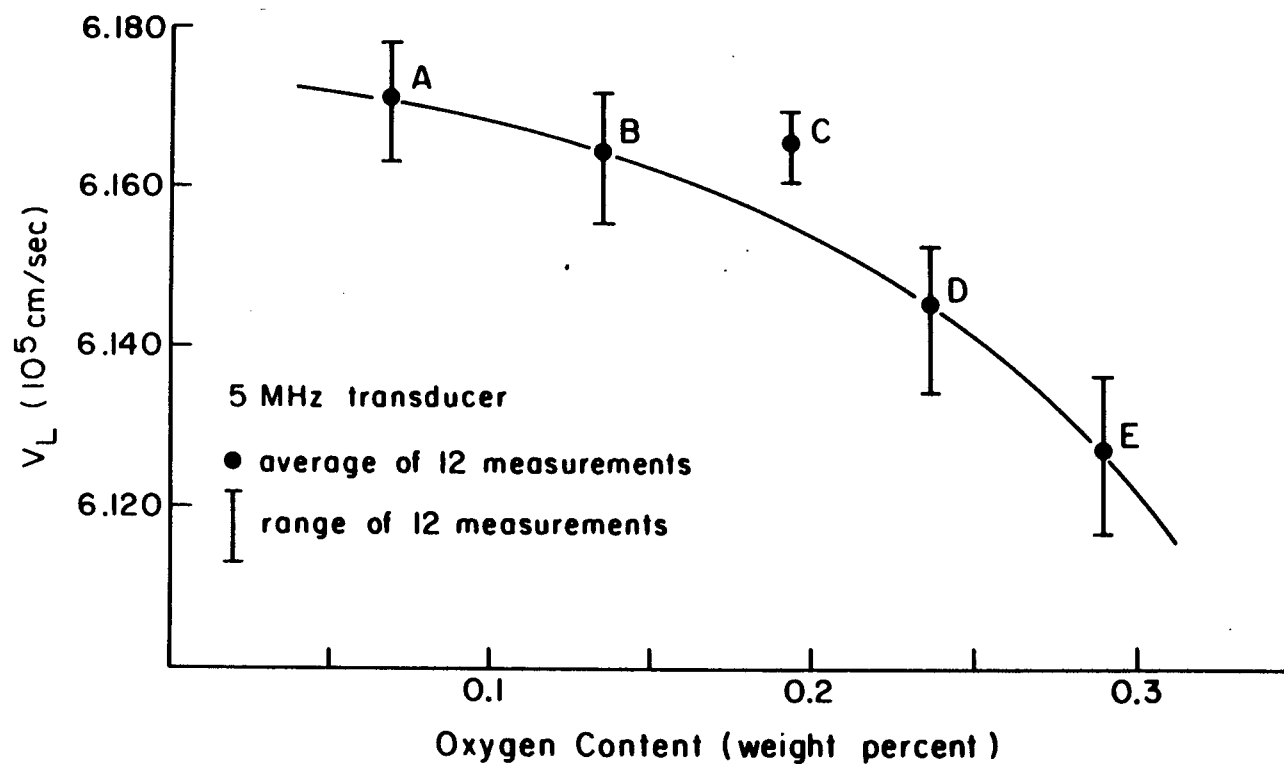


Fig. 4. Longitudinal Wave Velocity vs. Oxygen Content

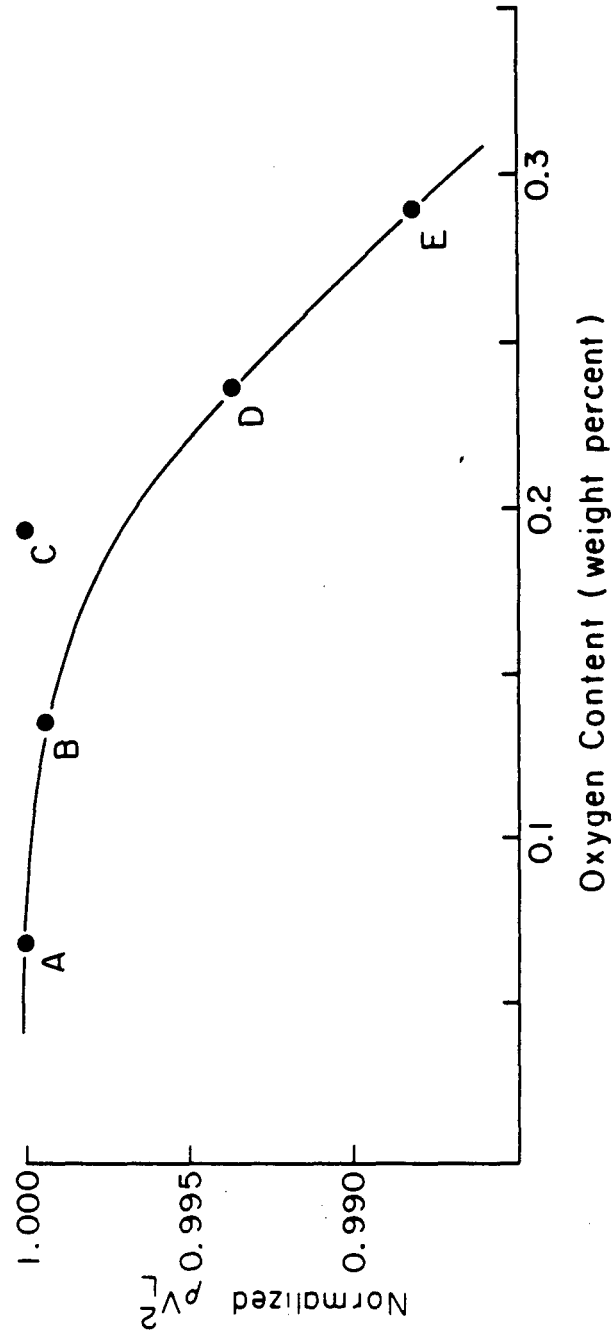


Fig. 5. Normalized  $\rho_V^2$  vs. Oxygen Content

specimens. As discussed earlier, Zener (5) theoretically demonstrated that the residual stress introduced by slight interstitial alloying in any metal necessarily reduces the elastic moduli. However, his theory was based on purely geometric considerations and did not consider the importance of the complex Ti-O electronic interaction. Additionally, Zener's conclusions were in conflict with the experimental results of Hsu and Conrad (18) who showed that in 100% alpha titanium, longitudinal wave velocity and, therefore, the elastic moduli, increased with increasing oxygen content. Hence, it is doubtful that the theoretical arguments of Zener (5) account for the results obtained here. In fact, if the only parameter changing was the amount of oxygen in the alpha-phase lattice of the Ti-6211 alloy, following Hsu and Conrad (18) we would expect  $V_L$  to increase with oxygen content rather than decrease.

A second factor that could be affecting the wave velocity is the relative amounts of the alpha and beta phases. The beta phase, the high temperature BCC phase, has a more open structure than the HCP alpha phase so one would expect a lower wave velocity in the beta phase material. If as a result of the thermomechanical processing the specimen received, increasing oxygen content somehow resulted in more retained beta phase material, then this could account for the observed behavior. However, both x-ray diffraction analysis and quantitative metallographic analysis failed to support the hypothesis and in fact indicated that, as would be expected, the amount of beta phase actually tended to decrease

with increasing oxygen content.

Other factors that could influence  $V_L$  would be differences in texture (29) and residual stress introduced by rolling the plate specimens with varying oxygen levels. The higher the oxygen content, the greater the resistance to rolling deformation would be, which in turn would cause larger residual compressive stresses and the decreasing wave velocity behavior observed. The contribution of residual stress can be studied by performing a stress relieving heat treatment on the specimens (assuming this heat treatment doesn't affect the texture and microstructure) and then repeating the ultrasonic wave velocity measurements. This set of experiments is currently underway.

#### Ultrasonic Attenuation Measurements

Attenuation measurements were performed on the oxygen contaminated specimens to evaluate the usefulness of attenuation for quantitatively detecting the presence of oxygen. The results, shown in Fig. 6, demonstrate the inherent scatter present in attenuation measurements using contact transducers due to the sensitivity of this technique. The elastomer coating used to couple the transducer to the specimen provided a fairly reproducible transducer specimen coupling and helped decrease the data scatter. Between 24 and 32 attenuation measurements were performed on each specimen; the average and range of these measurements are presented in Fig. 6. Statistical reduction of the attenuation data (see Fig. 7) indicates that ultrasonic attenuation may have potential for quantitatively detecting the presence of oxygen in Ti-6211. It is not known whether the

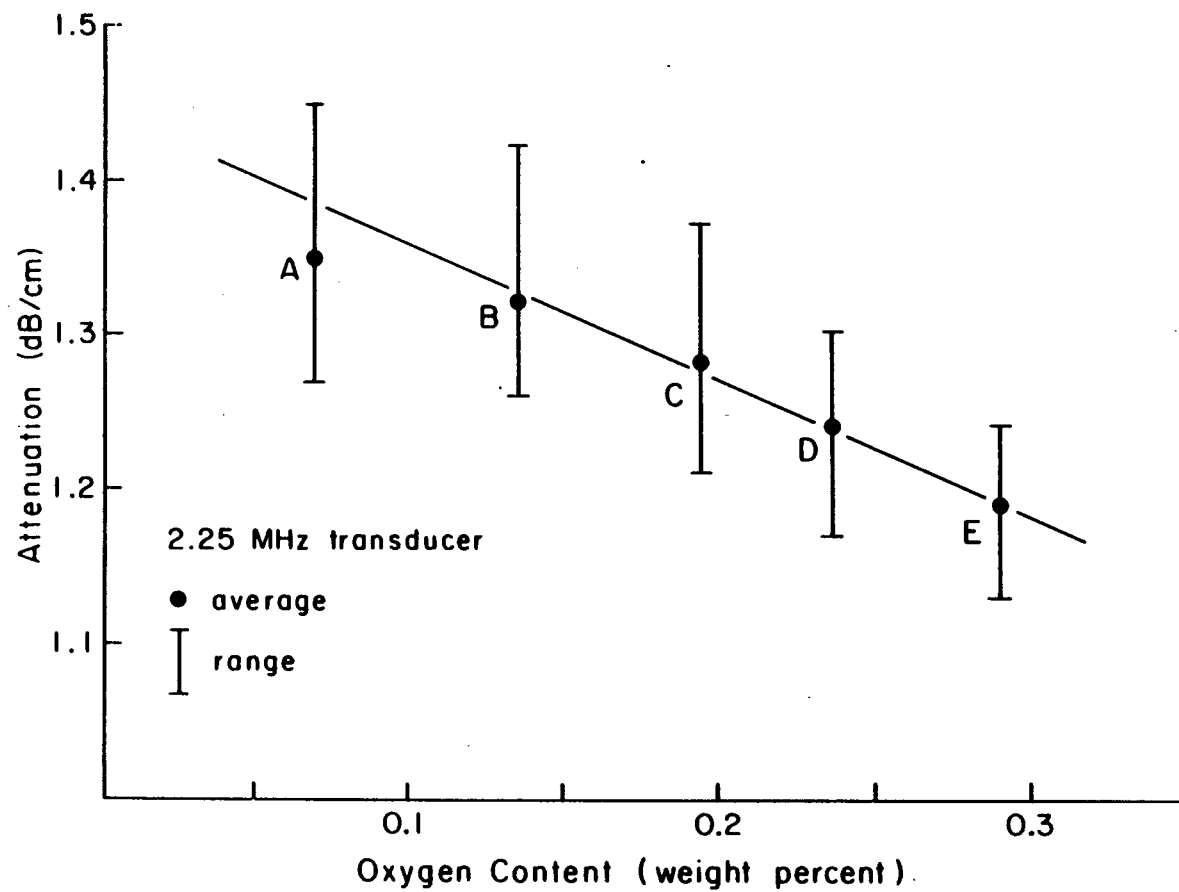


Fig. 6. Longitudinal Wave Attenuation vs. Oxygen Content

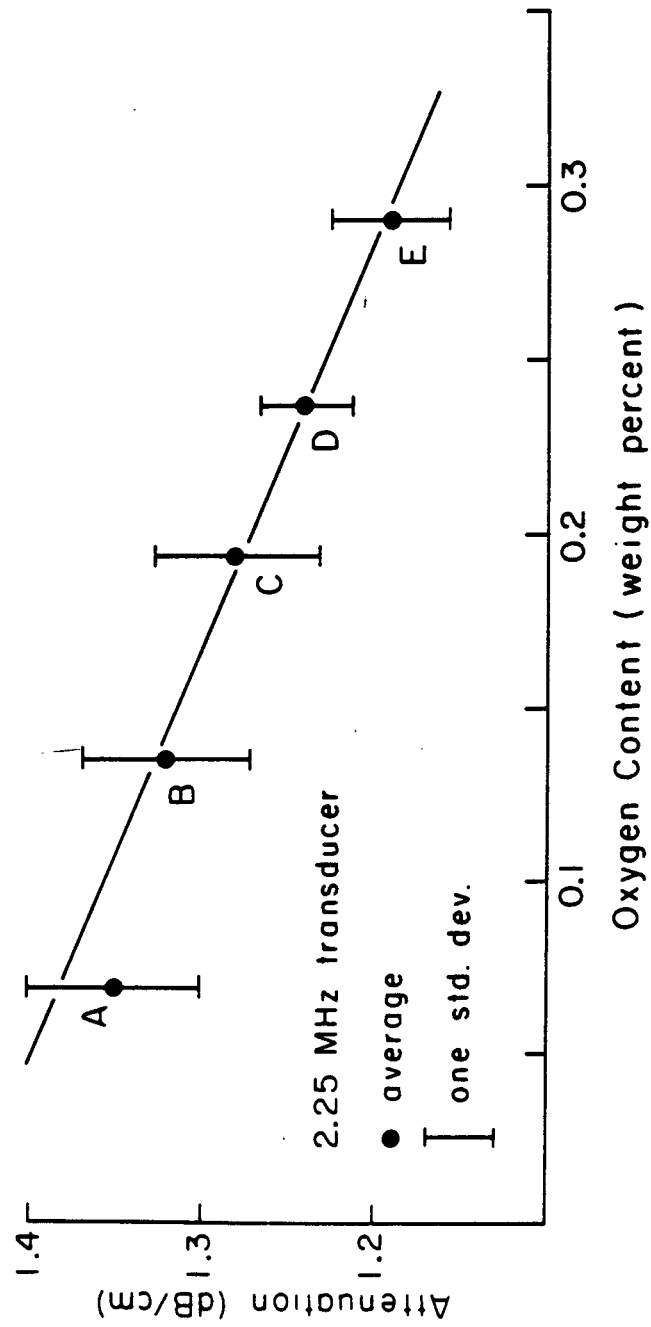


Fig. 7. Longitudinal Wave Attenuation vs. Oxygen Content

decrease in attenuation with increasing oxygen content is due to the effect of the oxygen on the alpha titanium lattice, the change in the amount of retained beta phase, or the microstructural variations induced by oxygen and thermal history.

#### Hardness Measurements

Rockwell "C" hardness, a measure of material response to plastic deformation, was found to increase with increasing oxygen content (see Fig. 8). The increase was probably due to the solid solution strengthening provided by the interstitial oxygen, which partitions preferentially into the alpha phase (4, 6, 7). This increase is consistent with results reported in the literature. An alternative explanation could involve the changing distribution of an alpha phase and beta phase material as the oxygen content changes.

#### CONCLUSIONS

Analysis of the data collected from the oxygen contaminated specimens has underscored some of the material effects such as high temperature phase retention, microstructural alteration, texture, and residual stress that influence the ultrasonic testing of the Ti-6211 alloy and metal alloys in general. Both ultrasonic wave velocity and ultrasonic attenuation were found to decrease with increasing oxygen content as a result of these material effects. Additionally, the variations in ultrasonic data were correlated with the results of scanning electron micros-

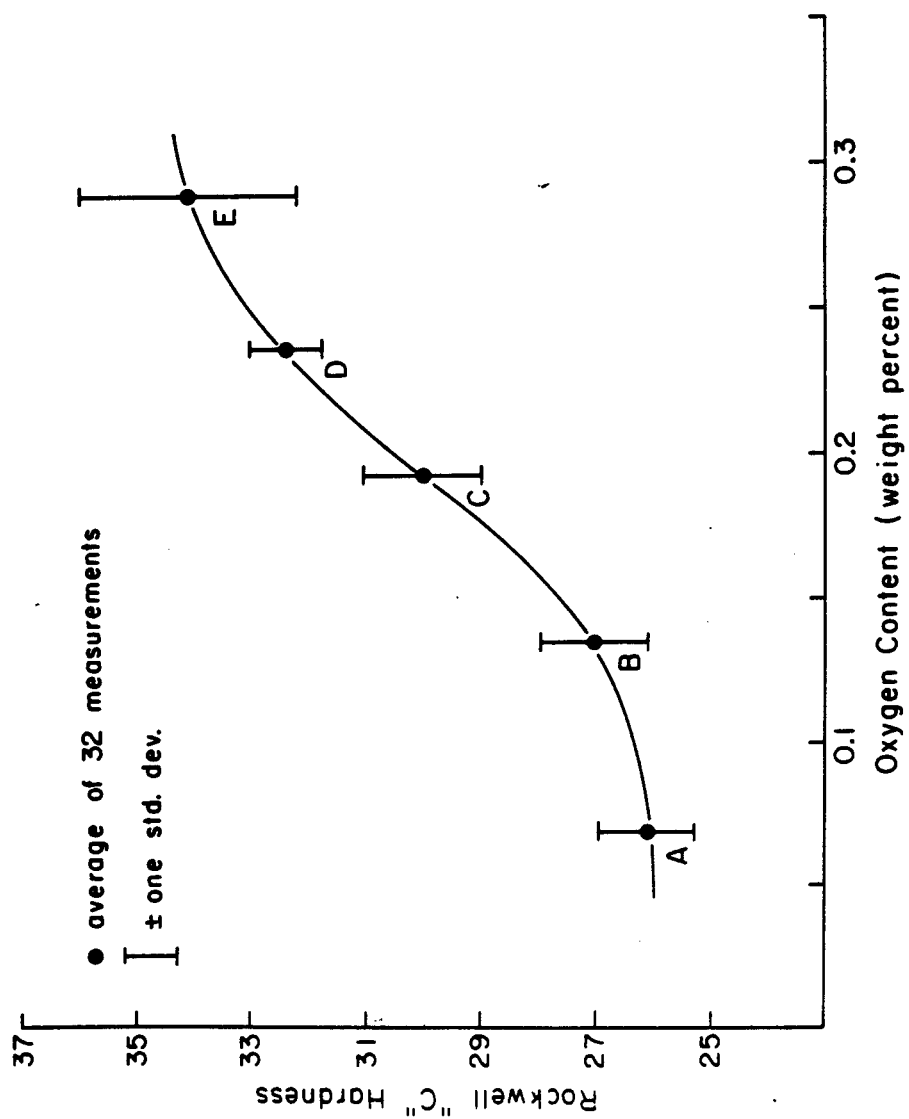


Fig. 8. Rockwell "C" Hardness vs. Oxygen Content



copy and hardness testing. This work also indicated that ultrasonic attenuation shows promise as a nondestructive tool for quantitatively evaluating the amount of oxygen present in a Ti-6211 sample.

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